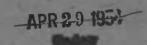
(200) T672 Mo. 412

AN AUTOMATIC MICROMUFFLE



FOR THE DETERMINATION OF

ASH IN CARBONACEOUS MATERIAL

By Robert Meyrowitz and C. J. Massoni

Trace Elements Investigations Report 412

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

ੂ**ሪ**ባ ንረ ሮ**ሪ**ዐ

Chemistry

This document consists of 7 pages. Series A.

UNITED STATES DEPARTMENT OF THE INTERIOR GEOLOGICAL SURVEY

AN AUTOMATIC MICROMUFFLE FOR THE DETERMINATION OF ASH IN CARBONACEOUS MATERIAL*

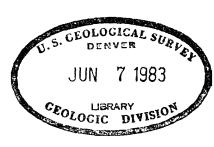
Ву

Robert Meyrowitz and C. J. Massoni

March 1954

Trace Elements Investigations Report 412

This preliminary report is distributed without editorial and technical review for conformity with official standards and nomenclature. It is not for public inspection or quotation.



^{*}This report concerns work done on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

,			
,	/		

USGS - TEI-412

CHEMISTRY

American Cyanamid Company, Winchester 1 Argonne National Laboratory 1 Atomic Energy Commission, Washington 2 Battelle Memorial Institute, Columbus 1 Carbide and Carbon Chemicals Company, Y-12 Area 1 Division of Raw Materials, Albuquerque 1 Division of Raw Materials, Butte 1 Division of Raw Materials, Butte 1 Division of Raw Materials, Denver 1 Division of Raw Materials, Douglas 1 Division of Raw Materials, Hot Springs 1 Division of Raw Materials, New York 6 Division of Raw Materials, New York 6 Division of Raw Materials, Richfield 1 Division of Raw Materials, Richfield 1 Division of Raw Materials, Washington 3 Division of Raw Materials, Washington 3 Division of Raw Materials, Washington 3 Division of Raw Materials, Washington 5 Division of Raw Materials, Washington 1 Dow Chemical Company, Pitteburg 1 Exploration Division, Grand Junction Operations Office 1 Grand Junction Operations Office 1 Grand Junction Operations Office 1 Grendssee Valley Authority, Wilson Dam 1 U. S. Geological Survey: Alaskan Geology Branch, Washington 1 Deals Company, Mashington 1 Deals Company, Mashington 1 Deals Cardner, Albuquerque 1 D. R. Cooper, Denver 1 D. R. Cooper, Denver 1 D. R. A. Laurence, Knoxville 1 D. M. Lemmon, Washington 1 D. M. Denson, Denver 1 D. A. H. Koschmann, Denver 1 D. A. Lewissenborn, Spokane 1 D. S. Grebres, Plant City 1 D. M. Schopf, Columbus 1 D. S. Grebres, Denver 1 D. A. E. Weissenborn, Spokane 1 D. T. Smith, Jr., Denver 1 D. A. E. Weissenborn, Spokane 1 D. C. Control of the Street School o	Distribution (Series A)	No.	of	copies
Argone National Laboratory	American Cyanamid Company Winchester		_ 7	!
Atomic Energy Commission, Washington 2 Battelle Memorial Institute, Columbus 1 Carbide and Carbon Chemicals Company, Y-12 Area 1 Division of Raw Materials, Albuquerque 1 Division of Raw Materials, Butte 1 Division of Raw Materials, Denver 1 Division of Raw Materials, Denver 1 Division of Raw Materials, Douglas 1 Division of Raw Materials, Hot Springs 1 Division of Raw Materials, New York 6 Division of Raw Materials, New York 6 Division of Raw Materials, New York 6 Division of Raw Materials, Richfield 1 Division of Raw Materials, Salt Lake City 1 Division of Raw Materials, Salt Lake City 1 Division of Raw Materials, Washington 3 Division of Raw Materials, Washington 1 Dow Chemical Company, Pittsburg 1 Exploration Division, Grand Junction Operations Office 1 Grand Junction Operations Office 1 Technical Information Service, Oak Ridge 6 Tennessee Valley Authority, Wilson Dam 1 U. S. Geological Survey: Alaskan Geology Branch, Washington 1 Fuels Branch, Washington 1 Fuels Branch, Washington 1 E. H. Bailey, Menlo Park 1 K. L. Buck, Denver 1 N. M. Denson, Denver 1 N. A. Isurence, Knoxville 1 D. M. Lemmon, Washington 1 J. D. Love, Laramie 1 R. G. Petersen, Plant City 1 R. J. Roberts, Salt Iske City 1 J. M. Schopf, Columbus 1 J. F. Sinth, Jr., Denver 1 R. W. Swanson, Spokane 1 TERCO, PRS, Washington 2				_
Battelle Memorial Institute, Columbus 1 Carbide and Carbon Chemicals Company, Y-12 Area 1 Division of Raw Materials, Albuquerque 1 1 1 1 1 1 1 1 1	· · · · · · · · · · · · · · · · · · ·			2
Carbide and Carbon Chemicals Company, Y-12 Area 1 Division of Raw Materials, Albuquerque 1 Division of Raw Materials, Butte 1 Division of Raw Materials, Denver 1 Division of Raw Materials, Denver 1 Division of Raw Materials, Douglas 1 Division of Raw Materials, Ishpeming 1 Division of Raw Materials, Ishpeming 1 Division of Raw Materials, Ishpeming 1 Division of Raw Materials, New York 6 6 Division of Raw Materials, Richfield 1 Division of Raw Materials, Richfield 1 Division of Raw Materials, Richfield 1 Division of Raw Materials, Salt Lake City 1 Division of Raw Materials, Salt Lake City 1 Division of Raw Materials, Washington 3 Division of Research, Washington 5 Division of Research, Washington 1 Dow Chemical Company, Pittsburg 1 Exploration Division, Grand Junction Operations Office 1 Grand Junction Operations Office 1 Technical Information Service, Oak Ridge 6 Tennessee Valley Authority, Wilson Dam 1 U. S. Geological Survey: Alaskan Geology Branch, Washington 1 Geochemistry and Petrology Branch, Washington 1 Geophysics Branch, Washington 1 Geophysics Branch, Washington 1 Division Deposits Branch, Washington 1 Division, Denver 1 Division Denver			-	
Division of Raw Materials, Albuquerque			-	
Division of Raw Materials, Butte 1 Division of Raw Materials, Denver 1 Division of Raw Materials, Douglas 1 Division of Raw Materials, Douglas 1 Division of Raw Materials, Ishpeming 1 Division of Raw Materials, Ishpeming 1 Division of Raw Materials, Phoenix 1 Division of Raw Materials, Phoenix 1 Division of Raw Materials, Phoenix 1 Division of Raw Materials, Richfield 1 Division of Raw Materials, Salt Lake City 1 Division of Raw Materials, Washington 3 Division of Rasearch, Washington 1 Division of Rasearch, Washington 1 Division of Research, Washington 1 Division of Research, Washington 1 Division of Research, Washington 1 Exploration Division, Grand Junction Operations Office 1 Division of Research, Washington 1 U. S. Geological Survey: Alaskan Geology Branch, Washington 1 Division Branch, Washington 2 Division Branch, Washington 2 Division Branch, Washington 2 Divisio				L
Division of Raw Materials, Denver				L.
Division of Raw Materials, Douglas 1	· · · · · · · · · · · · · · · · · · ·			L
Division of Raw Materials, Hot Springs	Division of Raw Materials, Douglas	0 0 0 0	.]	L
Division of Raw Materials, Ishpeming				ŀ
Division of Raw Materials, New York				_
Division of Raw Materials, Phoenix				_
Division of Raw Materials, Richfield 1 Division of Raw Materials, Salt Lake City 1 1 1 1 1 1 1 1 1			-	
Division of Raw Materials, Salt Lake City				
Division of Raw Materials, Washington				_
Division of Research, Washington				
Dow Chemical Company, Pittsburg				
Exploration Division, Grand Junction Operations Office				
Grand Junction Operations Office				
Technical Information Service, Oak Ridge	•			L
Tennessee Valley Authority, Wilson Dam				5
U. S. Geological Survey: Alaskan Geology Branch, Washington				L.
Alaskan Geology Branch, Washington				
Fuels Branch, Washington 1 Geochemistry and Petrology Branch, Washington 15 Geophysics Branch, Washington 1 Mineral Deposits Branch, Washington 1 E. H. Bailey, Menlo Park 1 K. L. Buck, Denver 1 J. R. Cooper, Denver 1 N. M. Denson, Denver 1 C. E. Dutton, Madison 1 R. P. Fischer, Grand Junction 1 L. S. Gardner, Albuquerque 1 M. R. Klepper, Washington 1 A. H. Koschmann, Denver 1 R. A. Laurence, Knoxville 1 D. M. Lemmon, Washington 1 J. D. Love, Laramie 1 R. G. Petersen, Plant City 1 R. J. Roberts, Salt Lake City 1 J. M. Schopf, Columbus 1 Q. D. Singewald, Beltsville 1 J. F. Smith, Jr., Denver 1 R. W. Swanson, Spokane 1 A. E. Weissenborn, Spokane 1 TEPCO, RPS, Washington 2 TEPCO, RPS, Washington 2	_ *			<u>L</u>
Geochemistry and Petrology Branch, Washington				L
Mineral Deposits Branch, Washington E. H. Bailey, Menlo Park L. Buck, Denver J. R. Cooper, Denver N. M. Denson, Denver C. E. Dutton, Madison R. P. Fischer, Grand Junction L. S. Gardner, Albuquerque M. R. Klepper, Washington A. H. Koschmann, Denver R. A. Laurence, Knoxville D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane 1 TEPCO, Penyer TEPCO, Penyer (Including master)				5
Mineral Deposits Branch, Washington E. H. Bailey, Menlo Park K. L. Buck, Denver J. R. Cooper, Denver N. M. Denson, Denver C. E. Dutton, Madison R. P. Fischer, Grand Junction L. S. Gardner, Albuquerque M. R. Klepper, Washington A. H. Koschmann, Denver I. D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane TEPCO, Denver TEPCO, RPS, Washington 2 TEPCO, RPS, Washington 2 Tepco, RPS, Washington				
K. L. Buck, Denver				L
J. R. Cooper, Denver	E. H. Bailey, Menlo Park	0000		
N. M. Denson, Denver C. E. Dutton, Madison R. P. Fischer, Grand Junction L. S. Gardner, Albuquerque M. R. Klepper, Washington A. H. Koschmann, Denver D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane TEPCO, Denver TEPCO, RPS, Washington (Including master)	K. L. Buck, Denver	0000	-	
C. E. Dutton, Madison R. P. Fischer, Grand Junction L. S. Gardner, Albuquerque M. R. Klepper, Washington A. H. Koschmann, Denver L. D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane (Including master)				
R. P. Fischer, Grand Junction L. S. Gardner, Albuquerque M. R. Klepper, Washington A. H. Koschmann, Denver R. A. Laurence, Knoxville D. M. Lemmon, Washington 1 J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane TEPCO, Denver TEPCO, RPS, Washington (Including master)	· · · · · · · · · · · · · · · · · · ·		-	
L. S. Gardner, Albuquerque M. R. Klepper, Washington A. H. Koschmann, Denver R. A. Laurence, Knoxville D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane TEPCO, Denver TEPCO, RPS, Washington (Including master)			-	
M. R. Klepper, Washington A. H. Koschmann, Denver R. A. Laurence, Knoxville D. M. Lemmon, Washington I. J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane TEPCO, Denver TEPCO, RPS, Washington (Including master)	R. P. Fischer, Grand Junction	0000		
A. H. Koschmann, Denver R. A. Laurence, Knoxville D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus L. J. M. Schopf, Columbus L. J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane TEPCO, Denver (Including master)				
R. A. Laurence, Knoxville D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus Q. D. Singewald, Beltsville J. F. Smith, Jr., Denver R. W. Swanson, Spokane A. E. Weissenborn, Spokane TEPCO, Denver (Including master)				
D. M. Lemmon, Washington J. D. Love, Laramie R. G. Petersen, Plant City R. J. Roberts, Salt Lake City J. M. Schopf, Columbus J. M. Schopf, Columbus L. J. F. Smith, Jr., Denver R. W. Swanson, Spokane L. R. Weissenborn, Spokane TEPCO, Denver TEPCO, RPS, Washington (Including master)				
J. D. Love, Laramie			-	
R. G. Petersen, Plant City			-	
R. J. Roberts, Salt Lake City				
J. M. Schopf, Columbus			-	
Q. D. Singewald, Beltsville			-	
J. F. Smith, Jr., Denver				
R. W. Swanson, Spokane			-	
A. E. Weissenborn, Spokane			-	
TEPCO, Denver				
TEPCO, RPS, Washington			_	
(Including master)			-	
			-	NACONID .

1		

CONTENTS

Pa	ge
Abstract	4
Introduction	14
Description	5
References	7
ILLUSTRATION	
Pa	ge
Figure 1. Front view of apparatus	6

·			

AN AUTOMATIC MICROMUFFLE FOR THE DETERMINATION OF ASH IN CARBONACEOUS MATERIAL

By Robert Meyrowitz and C. J. Massoni

ABSTRACT

An automatic micromuffle for the determination of the ash content of small samples of carbonaceous material is comprised of a furnace of the radiant-heating type with a direct reading pyrometer for temperature control. This furnace can be assembled with a minimum of labor using parts available from scientific laboratory apparatus companies.

INTRODUCTION

A study of the mineralogy and geochemistry of carbonaceous rocks is being carried out as part of a program of research on the geochemistry of uranium that the Geological Survey is conducting on behalf of the Atomic Energy Commission. To help solve certain phases of the problem, an organic microanalytical laboratory has been organized. The purpose of this paper is to describe an automatic microcombustion apparatus which has been designed and used in this laboratory for the determination of the ash content of small amounts of carbonaceous materials. Very often the amount of material available for analysis is small, necessitating the use of micromethods. Norton, Royer, and Koegel (1940) have shown that there is a saving of time without any loss of precision when using the microtechnique for ash determinations.

		,

Two automatic micromuffles have been described (Norton, Royer, and Koegel, 1940; Steyermark, 1951). These micromuffles have been made to order and require a great deal of machine-shop work in their construction. The micromuffle described by Norton, Royer, and Koegel (1940) has platinum heating coils and the temperature is regulated by varying the voltage using a variable autotransformer. Either one or two samples can be burned at one time; the furnace is stationary, and the tubes are drawn through the furnace. There are two rates of travel, the slowest speed being 2.5 cm per 10 min. Steyermark's micromuffle (1951) is heated by means of small nichrome wire and temperature regulation is by means of a variable autotransformer. Two samples can be burned at one time. In this apparatus the tubes are stationary and the furnace is moved. There is one rate of travel which is 2.5 cm per 10 min.

The advantage of the micromuffle described here is that it can be assembled with a minimum of labor using parts available from scientific laboratory apparatus companies.

DESCRIPTION

The micromuffle pictured in figure 1 consists of two parts: (1) a furnace drive, single speed (2.0 cm per 10 min.), electric, having adjustable limit stops to arrest automatically the advance of the furnace; Arthur H. Thomas Co., Philadelphia 5, Pa., Catalog No. 5683K, and (2) the moving, short-furnace section of an automatic, micro- and semimicro- combustion apparatus, Catalog No. S-21580, available from E. H. Sargent and Co., Chicago 30, Ill.

		•	

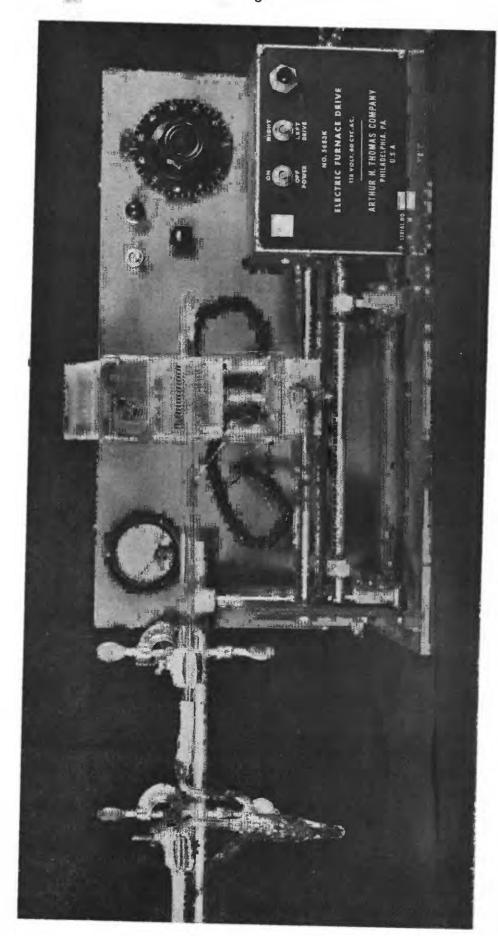


Figure 1.--Front view of apparatus.

		•	
		6	
*			
	-17	W 3 W	

This furnace is of the radiant-heating type, with hinged shell and open-sided heating elements and operates on 115 v, 60 cycle, a-c circuit. The furnace unit consists of six parts: (1) voltage reducing transformer, designed to operate on 100 v, 50/60 cycle, single phase service, the secondary is rated 10 v at 28 amps; (2) variable auto-transformer; (3) thermocouple and direct reading pyrometer for temperature control; (4) fuse; (5) pilot light; and (6) switch. The maximum temperature for continuous operation is 900 C.

The pyrometer is calibrated in the following manner. The thermocouple of a second pyrometer is inserted into the combustion tube until it reaches the position to be occupied by the combustion boat. While a stream of oxygen is flowing at the same rate to be used in the ash determination, the position of the thermocouple in the furnace is adjusted so that the reading of the pyrometer of the micromuffle is the same as that of the second pyrometer.

REFERENCES

- Norton, A. R., Royer, G. L., and Koegel, R., 1940, Ash in organic compounds. Determination by microtechnique with automatic combustion: Anal. Chemistry, v. 12, p. 121-123.
- Steyermark, Al, 1951, Quantitative organic microanalysis, Philadelphia, The Blakiston Company, p. 48-49.